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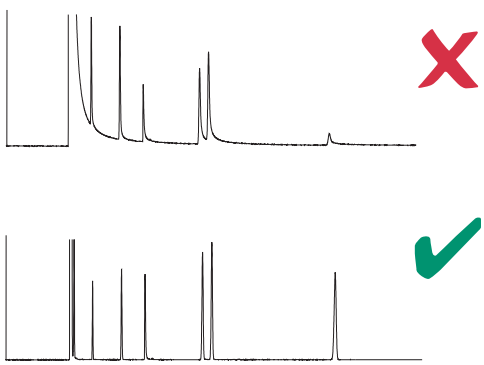
TECHNICAL ARTICLE

Useful tips for Column

Installation & Injection

Figure 1. From Bad to Good



Have you ever experienced a chromatogram that looks like the top one in **Figure 1**?
It is possible to go from the first chromatogram to the second, without changing the column and without cleaning the detector or injector.

INJECTION SPEED

The most common cause of split peaks is poor manual injection. Ideally for split injection, depress the syringe plunger as fast as possible, to get the sample into the liner as quickly as possible. A slow injection will increase peak width, adversely affecting efficiency and resolution. **Figure 2**

Figure 2. Effect of Injection Speed

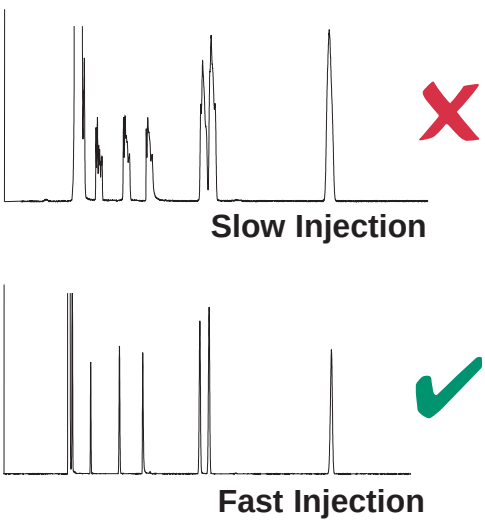
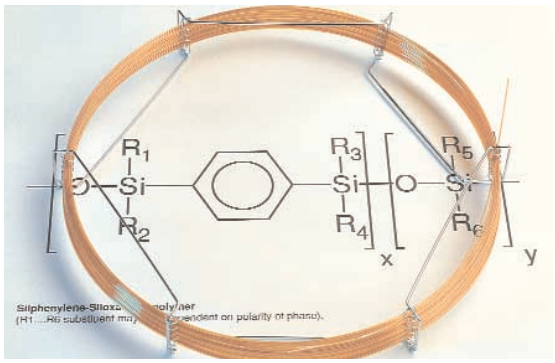


Figure 2 shows a slow injection (15 seconds) which results in split peaks, and a fast injection that demonstrates how good technique can improve results.



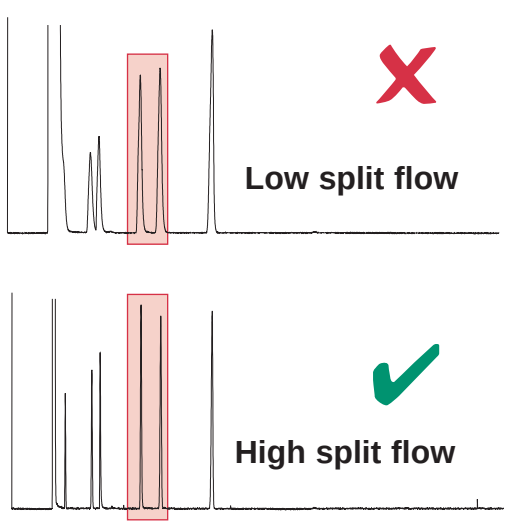
SPLIT FLOW

The two chromatograms in **Figure 3** emphasize the importance of adequate split flow.

In the top chromatogram, the split ratio is 50:1. The analysis has been carried out on a 0.1 mm ID column with a flow through the column of only 0.22 ml/min (27 cm/sec) at 33.2 psi. This would appear to be optimum conditions but the split flow is only 10.5 ml/min. (Oven temp is isothermal at 150°C.)

The second chromatogram shows the analysis using the same conditions, but the split flow has been increased to 50 ml/min (ratio = 250:1). Notice the dramatic improvement in the chromatography!

Figure 3. Effect of Split Flow

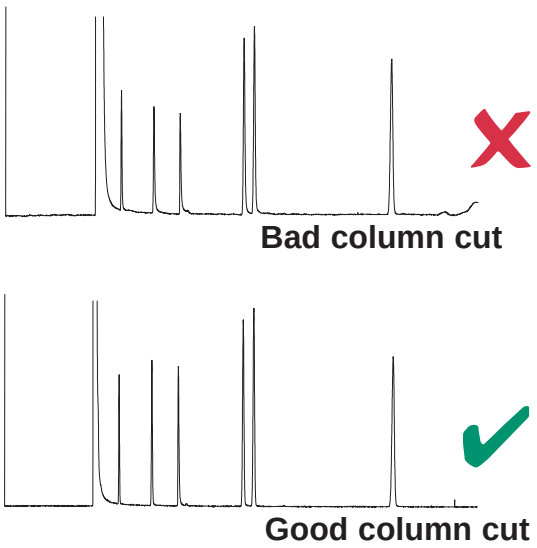


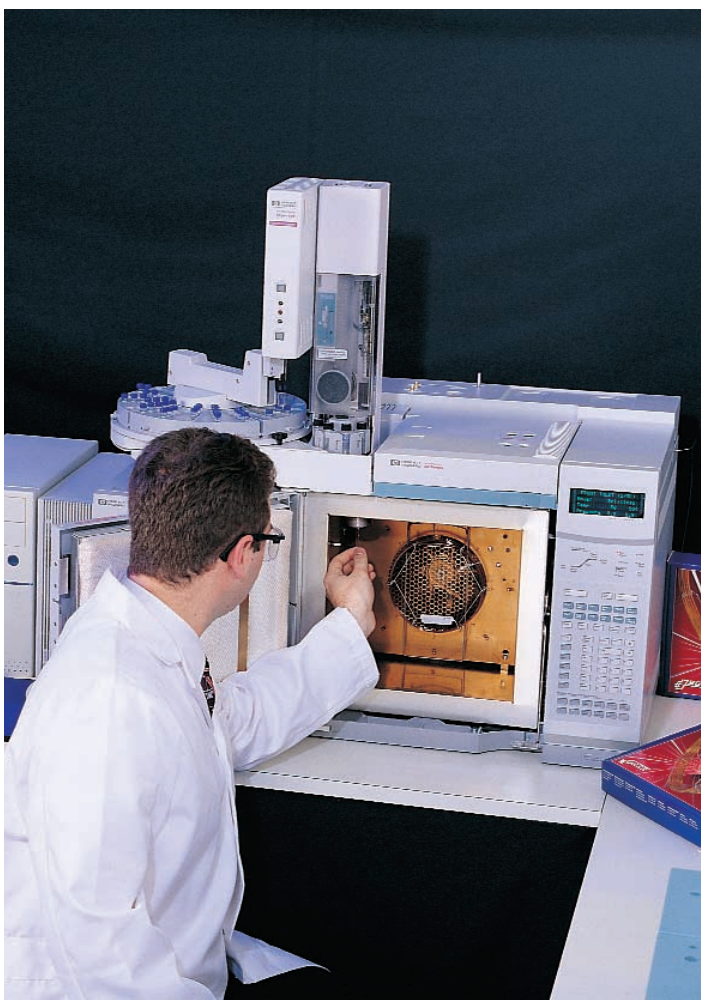
If the split flow is too low, there will be an increase in peak width (as seen in **Figure 3**), while at very high split flows, the residence time is not long enough for vaporization which can result in large mass discrimination.

COLUMN ENDS

Figure 4 shows the difference a clean, square cut can make to an analysis. Note the poorer solvent peak tailing with the bad column cut. A good column cut will also reduce the likelihood of blockages and ensure clean entry for on-column injection.

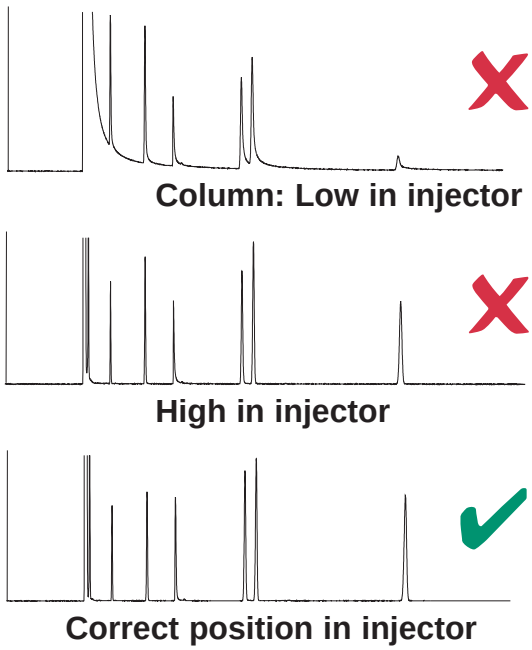
Figure 4. Effect of Column Cut





COLUMN INSTALLATION

Figure 5. Effect of Column Position



The three chromatograms in **Figure 5** show that the correct position of the column in the injection liner is critical. If the column is too low, all peaks will tail badly, even the hydrocarbons!

If the column is too high, peak shape appears to be OK but on closer examination there is mass discrimination occurring. (Mass discrimination is when there is non-quantitative transfer of the less volatile components compared with the volatile components.)

Figure 6 shows the correct position of the column in the injector.

Figure 6. Column Position in the Injector